23 SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE SPECTROMETRY (SEM-EDS)

23.1 Introduction to Scanning Electron Microscopy (SEM)

23.1.1 Objectives

Through completion of this module the trainee will develop the theoretical knowledge to be conversant in:

- The theory of SEM design and operation;
- The history and development of advances in SEM;
- The capabilities and limitations of the instrument; and,
- The QA/QC of the instrument.

23.1.2 Required Readings

23.1.2.1 Flegler, S. L., Heckman, J. W. and Klomparens, K. L., Scanning and Transmission Electron Microscopy An Introduction, Oxford University Press, 1993.

23.1.2.2 Gabriel, Barbara L., SEM: A User's Manual for Material Science, American Society for Metals, 1985.

23.1.2.3 Goldstein, J. I., Yakowitz, H., Newbury, D. E., Lifshin, E., Colby, J. W., and Coleman, J. R., Practical Scanning Electron Microscopy, Plenum Press, 1975.

23.1.2.4 Goldstein, J. I., et.al., Scanning Electron Microscopy and X-Ray Microanalysis, Plenum Press, 1981.

23.1.2.5 Postek, Michael T., et.al., Scanning Electron Microscopy: A Student's Handbook, Ladd Research Industries, Inc. 1980.

23.1.3 Questions

The trainee will provide written answers to the following questions:

- Give definitions for the following: depth of field; working distance; resolution.
- Describe the relationship to the items listed above with changes in accelerating voltage; objective aperture size and backscatter electron image.
- Describe how magnification is achieved in the SEM.
- What is lens hysteresis and why is it important?
- Compare and contrast electron gun sources.
- Describe the various signals produced in the SEM, how they are detected and what they are used for.
- Describe electron beam specimen interactions.
- Describe the vacuum systems used in the SEM.

23.1.4 Practical Exercises

23.1.4.1 The trainer will demonstrate the operation of the instrument to which the trainee will initially/primarily be assigned.

23.1.4.2 The trainee will correct an astigmatic image.

23.1.4.3 The trainee will demonstrate filament replacement, saturation and column liner replacement.

23.1.4.4 The trainee will demonstrate image capture and storage procedures.

23.1.4.5 The trainee will perform the monthly QC for the instrument to which they are assigned.

23.1.5 Evaluation

23.1.5.1 The trainer will review the written answers to the questions with the trainee.

23.1.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

23.1.5.3 Review of practical exercises.

23.1.5.4 The trainee will be quizzed upon the subject matter.

23.2 Introduction to Energy Dispersive Spectroscopy

23.2.1 Objectives

Through completion of this module the trainee will have developed and demonstrated theoretical knowledge and/or practical skills in:

- The theory of EDS design and operation;
- The history and development of advances in EDS;
- The capabilities and limitations of the instrument; and,
- The QA/QC of the instrument.

23.2.2 Required Readings

23.3.2 Multimedia Tutorial, The Principles and Practice of X-ray Microanalysis, Vols. 1 and 2, Oxford Instruments plc, 1997.

23.2.3 Questions

The trainee will provide written answers to the following questions:

- Describe the Bohr atomic model and how characteristic X-rays are named.
- Define escape peak, sum peak and system peak; what causes them and how you minimize them.
- Describe the components of the energy dispersive X-ray system.
- What is bremstrählung?
- What would an EDS spectrum be expected to look like if steric hindrance was a problem?
- How does “process time” affect spectral resolution? What are the advantages of increasing or decreasing process time?
- What is “dead time”? What happens if it becomes excessive?
- Define critical excitation energy. When is it appropriate to use low vs. high KV?
- What is meant by EDS resolution? What criteria is necessary to specify a specific element?
- Describe peak overlaps and specifically how to deal with Pb/S/Mo; Ti/Ba; Ca/Sb; P/Zr?
- What is zero offset and gain?
- What is the approximate detection limit for an EDS system?
- What is the difference between quantitative analysis and qualitative analysis?
- What is ZAF?
- Why is N more difficult to detect with a light element detector than C or O?

23.2.4 Practical Exercises

23.2.4.1 The trainer and trainee will prepare and analyze primer residue samples.

23.2.5 Evaluation

23.2.5.1 The trainer will review the written answers to the questions with the trainee.

23.2.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

23.2.5.3 Review of practical exercises.

23.2.5.4 The trainee will calibrate the instrument and demonstrate proper QA/QC, laboratory safety and equipment maintenance and operation techniques.

23.3 Instrument Support Specimen Preparation and Analysis

23.3.1 Objectives

Through completion of this module the trainee will have developed and demonstrated theoretical knowledge and/or practical skills to:

- Prepare instrument support samples for SEM-EDS analysis;
- Determine if a sample requires carbon coating;
- Understand how to prepare samples by freeze drying or gold sputter coating; and,
- Explain the appropriate approach and common pitfalls to data interpretation.

23.3.2 Required Readings

23.3.2.1 Henson, M. Lynn and Jergovich, Tammy A., “Scanning electron microscopy and energy dispersive X-ray spectrometry (SEM/EDS) for the forensic examination of paints and coatings”, Forensic Examination of Glass and Paint Analysis and Interpretation, Caddy, Brian, ed., Taylor and Francis, New York, 2001, Chapter 11, pp. 243-272.

23.3.2.2 Operators manual for Carbon evaporator.

23.3.2.3 Stromberg, Maehly, Chemical Criminalistics, O. Brandstetter: Wiesbaden, Germany, 1981, pp. 185-200.

23.3.2.4 Ward, Dennis C., and Carlson, Timothy L., "Paint Analysis Using the Scanning Electron Microscope," Crime Laboratory Digest, F.B.I. Laboratory, Washington, DC, 1983, pp.2-6.

23.3.3 Questions

The trainee will provide written answers to the following questions:

- What is “charging” and how can it be avoided?
- What are the advantages and disadvantages of gold sputter coating?
- Is the secondary image or backscatter image more useful when analyzing multilayered paint samples?
- How do homogeneity and heterogeneity in instrument support samples affect the data?
- How does the size of the area sampled affect the data?
- What is composite sampling and when might it be appropriate?

23.3.4 Practical Exercises

23.3.4.1 The trainer will demonstrate the complete operational cycle, to include proper clean up, of the carbon evaporator.

23.3.4.2 The trainer will observe the trainee complete a complete operational cycle, to include proper clean up, of the carbon evaporator.

23.3.4.3 The trainer and the trainee will discuss spectrum labeling techniques including all visible peaks to be labeled in an auto-scaled spectrum and the appropriate use of manual labels for escape peaks, sum peaks and peaks that would otherwise be illegible if computer labeling was used.

23.3.4.4 The trainer and the trainee will prepare and analyze paint instrument support samples to include as a minimum: multilayered samples, two-layered samples in cross-section and top/bottom, and smears.

23.3.4.5 The trainer and the trainee will prepare and analyze explosives and general chemical instrument support samples to include as a minimum whole powders and dried extracts.

23.3.4.6 The trainer and the trainee will prepare and analyze a tissue sample from an electrocution case, if available.

23.3.5 Evaluation

23.3.5.1 The trainer will review the written answers to the questions with the trainee.

23.3.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

23.3.5.3 Review of practical exercises.

23.4 Competency Evaluation and Mock Trial

The trainee will use SEM-EDS when completing their subdiscipline competency test and will defend their results as a part of their mock trial in that subdiscipline.

23.5 Reading List

23.5.1 Caddy, Brian, Ed., Forensic Examination of Glass and Paint Analysis and Interpretation, Taylor and Francis, New York, 2001.

23.5.2 Flegler, S. L., Heckman, J. W. and Klomparens, K. L., Scanning and Transmission Electron Microscopy An Introduction, Oxford University Press, 1993.

23.5.3 Gabriel, Barbara L., SEM: A User's Manual for Material Science, American Society for Metals, 1985.

23.5.4 Goldstein, J. I., Yakowitz, H., Newbury, D. E., Lifshin, E., Colby, J. W., and Coleman, J. R., Practical Scanning Electron Microscopy, Plenum Press, 1975.

23.5.5 Goldstein, J. I., et.al., Scanning Electron Microscopy and X-Ray Microanalysis, Plenum Press, 1981.

23.5.6 Multimedia Tutorial, The Principles and Practice of X-ray Microanalysis, Vols. 1 and 2, Oxford Instruments plc, 1997.

23.5.7 Operators manual for Carbon evaporator.

23.5.8 Postek, Michael T., et.al., Scanning Electron Microscopy: A Student's Handbook, Ladd Research Industries, Inc., 1980.

23.5.9 Stromberg, Maehly, Chemical Criminalistics, O. Brandstetter: Wiesbaden, Germany, 1981.

23.5.10 Ward, Dennis C., and Carlson, Timothy L., "Paint Analysis Using the Scanning Electron Microscope," Crime Laboratory Digest, F.B.I. Laboratory, Washington, DC, 1983.

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